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WATERTOWN ARSENAL LABORATORY

MEMORANDUM REPORT

NO. WAL 154/12

THE TEMPERING OF MARTENSITE

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> > DATE

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Watertown Arsenal Laboratory Memorandum

Report Number 154/12

Problem Number H-6.2a

THE TEMPERING OF MARTENSITE

Object

To determine by means of x-ray diffraction technique the time-temperature relations for the initial tempering of martensite.

Abstract

The time-temperature relation for the loss of tetragonality of martensite was determined for a plain carbon steel (.95% C.). The time and temperature are related through a heat of activation relation. This heat of activation is of the order of 34,000 cal./gm. mol. These data indicate that appreciable tempering of the first formed martensite can occur with moderate cooling rates upon quenching if the M_S temperature is significantly above room temperature. Graphs are presented for estimating the amount of tempering, in the steel studied, during any tempering treatment, as well as during quenching.

Introduction

In several earlier studies of quenched steel specimens, microstructures were observed containing a constituent identified as either bainite or tempered martensite. There has been some controversy as to whether martensite can temper appreciably on cooling, during the quench, from the temperature at which it forms to room temperature. In order to determine whether appreciable tempering can take place under these conditions and so account for the observed microstructures, it was proposed to determine the effect of time and temperature upon the progress of the initial stage of tempering. This stage consists of precipitation of carbide* from the martensite, and in steels containing more than about .25%

^{*}As has been discussed2, there is still some question as to the exact form of the initial precipitate.

carbon is associated with a decrease in the tetragonality of the martensite lattice structure, as measured at room temperaturel, 2.

Furthermore, it was believed that the stabilizing of austenite (with respect to the martensite reaction), which occurs gradually in mix-tures of austenite with martensite, might be associated with the tempering of the martensite already formed. If the relation between the time and temperature for the initial tempering should be found to be the same as for the stabilizing effect, a good possibility of a connection would be established. The time-temperature relation for the initial tempering is one of the two relations needed. The other is the time-temperature relation for the stabilizing effect; work on this is now under way.

As the initial stage of tempering is associated with little change of hardness, hardness could not well be used as a criterion of the degree of tempering*. Since the characteristic loss of tetragonality at room temperature accompanies the initial stage, it appeared that the proments of the tempering could be followed by x-ray diffraction measurements of the tetragonality.

Materials and Procedure

Discs 3/16" thick were cut from a bar of a plain carbon steel containing .95% carbon and .26% manganese. The specimens were ground concave on one side in order to fit the curvature of the x-ray camera. Before heat treatment the specimens were plated with a thin layer of nickel to reduce surface decarburization during heat treatment. The specimens were austenitized for 30 minutes at 900° C. in a nitrogen atmosphere and quenched in water to room temperature. They were then ground slowly to remove the nickel layer. The specimens were etched electrolytically for 30 minutes in a dilute HCl - H2SO4 solution at very low current density. Extreme care was taken to prevent any heating of the specimens during the removal of the surface. The specimens were then quenched in liquid nitrogen. X-ray measurements were made on all specimens to determine the original axial ratio of the tetragonal martensite. Following this measurement all specimens were stored in dry ice until they were tempered. One specimen was used for each tempering temperature. The specimen was tempered, for one time, cooled to room temperature for x-ray measurements, and then retempered for additional times. The tempering was accomplished in liquid baths controlled to within $\pm 2^{\circ}$ C.

In order to find the axial ratio, it was necessary to measure the interplaner distance for the (110) and the (101) planes of martensite. A Phragmen No. 2 Camera was used with unfiltered chromium radiation to

^{*}It is for this reason that the previous publication3 throws little light upon the initial stage.

record the diffracted beams from these planes. The effects of possible variables of experiment and measurement were minimized by superimposing a standard reference x-ray line on each film. The line used was that from the (200) plane of platinum. The platinum was dusted on the face of each specimen before exposure to the x-radiation. The distances on the film between the Ker line diffracted from the (200) plane of platinum and the lines from both the (110) and (101) planes of the martensite were measured on a densitometer. Three measurements were made on each film of the distance between the densest portions of the lines. The measurements were averaged, and the c/a ratio calculated from the average as described in Appendix A. The measured distances and the calculated interplaner spacings and c/a ratios are given in Table I.

On some films the distance between the platinum line and the martensite (110) line could not be read satisfactorily. In these cases, the interplaner spacing, d, for the (110) planes was taken as equal to the average of the d(110) for other films taken of the same specimen. This procedure is believed justified because the d(110) was found to change very little as tempering proceeded (Table I). This is in accord with the published finding that the axial length a, to which d(110) is proportional (Appendix C), changes very little with changes in carbon content of the martensite.

Results and Discussion

The results are given in Table I, and plotted in Figure 1. In this figure the loss of tetragonality is expressed as a percentage of the total possible decrease of the c/a ratio, from the untempered value to c/a = 1. (a is the length of the two equal short axes of the tetragonal unit cell, while c is length of the long axis.)

Combinations of tempering temperature and tempering time that give the same loss of tetragonality were rend off the curves of Figure 1, and are plotted in Figure 2. Figure 2 indicates that the loss of tetragonality follows a heat of activation relationship, with a heat of activation of approximately 34,000 calories per gram-mol. That is,

$$tetragonality = f(N), (1)$$

where the parameter

$$N = te^{-q/RT} = te^{-34,000/1.986T} = t \cdot 10^{-7340/T}$$
 (2)

Here

t = tempering time

T = tempering temperature in °K.

q = heat of activation

R = universal gas constant.

In Figure 3, the loss in tetragonality is plotted as a function of the parameter N.

The rate of tempering at room temperature is of considerable practical interest. Figure 3 indicates that at a temperature of 20° C. the tetragonality will decrease 10% in about 5 months, 20% in about 5 years, 30% in 20 years, and 50% in 100 years.

To evaluate the amount of tempering that takes place during quenching, Equation 2 is put in the integral form,

$$N = \int_0^t e^{-q/RT} dt = \int_0^t 10^{-7340/T} dt,$$
 (3)

t being measured from the instant at which the martensite formed. It is also necessary to specify the relationship between temperature and time during the quench. A relation that is widely applicable is:

$$T = T_1 + (T_0 - T_1)e^{-ct},$$
 (4)

where

 T_1 = temperature of the quenching medium, T_0 = steel temperature at t = 0, c = cooling coefficient, a constant.

This exponential relationship is approached at low temperatures in almost all cases. Using Equations 3 and 4, Figure 4 has been derived, as indicated in Appendix D, for the case of a quenching medium at room temperature (20° C.). This figure gives the tempering parameter N obtained by exponential cooling to essentially room temperature as a function of the temperature at which the martensite forms and of the cooling coefficient c. The cooling coefficient c for simple shapes is given by the following equations:

Plates:
$$c = (HD)4a/D^2 = 4aH/D$$
,
Cylinders: $c = 2(HD)4a/D^2 = gaH/D$, (5)
Spheres: $c = 3(HD)4a/D^2 = 12aH/D$.

Here

H = severity of quench,
D = diameter or thickness,
a = thermal diffusivity of the steel.

More complicated shapes may be considered as composed of several simple shapes, and the c for the combination obtained by adding the c's for the component simple shapes. Thus, a short round of diameter d and length L can be considered a combination of a cylinder of diameter d and a plate of thickness L. The value of c for the round is then:

 $c = \frac{8aH}{d} + \frac{4aH}{L}.$

Since the thermal diffusivity, a, of martensite is of the order of .Ol inch²/second, Equations 5 may be roughly taken as:

Plates: c = .04H/D, Cylinders: c = .08H/D, Spheres: c = .12H/D. (6)

for dimensions expressed in inches and times in seconds. The quenching severity H in the martensite temperature range may vary from the order of .01 inch-1 for still air to the order of 10 inch-1 for agitated water.

A few examples may prove useful. A steel sheet .04 inch thick quenched in violently agitated water with H = 10 inch⁻¹ would have a cooling coefficient, c, according to Equation 6, of (.04)10/(.04) = 10 seconds⁻¹, or a log c of 1. Martensite forming at 300° C. has, by the time it approaches room temperature, tempered to the extent that log c + log N = -14, according to Figure 4. In the case mentioned, log N would then equal -15. Figure 3 shows that, if the sheet were made of steel that tempers, initially, as fast as the steel studied experimentally, considerably more than 50% of the tetragonality would be lost before the martensite formed at 300° C. reached approximately room temperature. Martensite formed at 200° C. has a log c + log N = -16.6. For the extremely rapid cooling mentioned, log N, therefore, equals -17.6. The martensite formed at 200° C. would lose 10-15% of its tetragonality by the time it reached room temperature.

As an example of extremely slow cooling, consider a 10 inch thick plate cooling in still air with H=.01 inch-1. Here $\log c=.4.4$, Martensite formed at 100° C., where $\log c + \log N = -20.6$, would temper to $\log N = -16.2$, and so lose about 35% of its tetragonality.

An another example, the air-cooled end of a standard Jominy endquench hardenability specimen may be considered. The cooling coefficient in the martensite range can be estimated to be of the order of .01 second⁻¹, so $\log c = -2$. Martensite formed at 200° C. would temper to $\log N = -14.6$, and most, if not all, of the tetragonality would be lost.

Finally, in the experimental work described in this report, the portion of the specimen used for diffraction measurements was about 1/8 inch thick when quenched. The quenching severity in the martensite range might be of the order of 4 inch-1. The cooling coefficient was, therefore, about 1.3 second-1 and log c about 0. The Mg temperature of the steel used may be estimated as approximately 200° C. Martensite formed just below the Mg temperature would, therefore, lose about 25% of its tetragonality by the time it reached room temperature. With an Mg of 200° C., most of the martensite would form below 150° C. This martensite would lose less than 10% of its tetragonality. Thus, while some tempering had undoubtedly occurred in the nominally untempered specimens, most of the martensite had tempered only slightly. The resulting variation

in tetragonality within the specimens accounts for at least some of the observed broadness of the diffraction lines.

The establishment of the heat of activation for the initial stage of tempering gives a little information concerning the rate of diffusion of carbon through ferrite, which has never been measured. Since the initial stage of tempering undoubtedly involves diffusion of carbon through martensite, it can readily be shown that the heat of activation for this stage of tempering must be more than the heat of activation for the diffusion. The heat of activation for the diffusion of carbon through (supersaturated) ferrite (martensite) is therefore, less than 34,000 calorics per gram-mol. This accords with the deduction, from the lattice structures of ferrite and austenite, that the heat of activation for the diffusion of carbon in ferrite must be less than the 36,000 calories per gram-mol measured for the diffusion of carbon in austenite.

The loss of tetragonality of the martensite may appear to be a somewhat arbitrary measure of tempering. However, it appears to be generally accepted that, as long as the martensite is tetragonal, the tetragonality (C - 1) is approximately proportional to the carbon content of the martensite.

The relation between time and temperature for the initial stage of tempering, discussed above, is based upon experimental results on only one steel, and its generality has, therefore, not been established. However, consideration of the nature of the initial stage seems to indicate that the progress of the reaction, and particularly the heat of activation, will probably not be greatly affected by changes in carbon content, and will probably be affected very little by changes in alloy content.

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MM 11/1/45

TABLE I

DATA AND RESULTS

Film No.	Tempering Time in Min.	X* in mm.	X ₁ ** in mm.	đ(110)	d(101)	c a	% Possible Decrease in C
		<u>60</u>	o C. Tempe	ring Tempe	rature		
T2 T2-1 T2-2 T2-3 T2-4 T2-5	0 1 10 100 1000	4.297 4.118 4.114 4.112 4.127	6.827 6.577 6.554 6.411 6.252 5.826	2.017 2.015 2.015 2.015 2.015	2.055 2.051 2.049 2.046 2.040	1.038 1.037 1.037 1.034 1.032	0 2.63 2.63 10.53 15.79 31.58
		<u>91</u>	O C. Tempe	ring Tempe:	rature		
T10-3 T10-4 T10-5 T10-6 T10-7 T10-8	0 2.5 8.0 25.0 80.0 250.0 800.0	4.227 4.369 4.291 4.163	6.748 6.353 6.548 6.404 6.171 5.962 5.372	2.016 *** 2.017 2.017 2.015 ***	2.054 2.047 2.051 2.049 2.044 2.042 2.033	1.039 1.032 1.034 1.032 1.030 1.027	0 17.94 12.82 17.94 23.08 30.77 56.41
109° C. Tempering Temperature							
T18-1 T18-2 T18-3 T18-4 T18-5 T18-6 T18-7	0 .25 .80 2.50 8.0 25.0 80.0 250.0	4.224 4.292 4.251 4.317 4.680	6.629 6.544 6.417 6.266 6.380 5.730 5.483 5.257	2.016 2.017 2.016 2.017 2.022 *** ***	2.052 2.051 2.049 2.046 2.048 2.038 2.035 2.031	1.037 1.034 1.033 1.030 1.026 1.022 1.020	0 8.10 10.81 18.92 29.73 40.54 45.95 59.46
139° C. Tempering Temperature							
T16 T16-1 T16-2 T16-3 T16-4	0 •25 •80 2.50 8.00	4.143	6.696 6.282 5.916 5.616 5.332	2.015 *** *** ***	2.053 2.046 2.041 2.037 2.033	1.040 1.032 1.027 1.022 1.019	0 20.00 32.50 45.00 52.50

^{*}X = distance between Platinum line and Martensite (110) line.

^{**}X1= distance between Platinum line and Martensite (101) line. ***Where Martensite (110) line was unreadable a d(110) value equal to the average obtainable value for previous readings was used in calculating the c ratio.

TABLE I (CONT.)

Film No.	Tempering Time in Min.	X* in mm.	X** in mm.	d(110)	d(101)	<u>c</u>	% Possible Decrease in Ca
T14 T14-2 T14-3 T14-4	0 .8 2.5 8.0	4:.093 	6.596 5.469 5.164 4.494	2.014 *** ***	2.051 2.034 2.030 2.026	1.039 1.020 1.016 1.012	0 48.72 58.97 69.23

^{*}X = distance between Platinum line and Martensite (110) line.

**X1 = distance between Platinum line and Martensite (101) line.

***Where Martensite (110) line was unreadable a d(110) value equal to the average obtainable value for previous readings was used in calculating the Tatio.

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APPENDIX A

METHOD OF CALCULATION

From the measured distances on the film between the platinum (200) line and the martensite (110) and (101) lines, the diffraction angles for the martensite lines are calculated by the following equation, derived in Appendix B:

$$\theta_{\rm m} = 35.717 - .28364X.$$
 (A1)

Here

\theta_m = Bragg diffraction angle, in degrees, for martensite line,
 X = measured distance on film, in millimoters.

From the diffraction angles, the spacing of the martensite lattice planes, d, was computed as:

$$d = \frac{\lambda}{2 \sin \theta} \tag{A2}$$

This equation is obtained directly from Bragg's Law. The wavelength \(\lambda \) was taken, for chromium Kal radiation, as 2285.0 Siegbahn units*.

To obtain the c/a ratio, the d values were substituted in the equation.

$$c/a = \sqrt{\frac{d_2^2}{2d_1^2 - d_2^2}}.$$
 (A3)

This equation is derived in Appendix C. d_1 represents the spacing of the (110) plane and d_2 the spacing of the (101) plane.

^{*}The c/a ratio could have been determined directly from the diffraction angles without calculating the lattice spacing, but it was convenient to have the spacing values.

APPENDIX B

RELATION BETWEEN FILM MEASUREMENTS AND DIFFRACTION ANGLE

Since an angle formed by a chord and a tangent of a circle is measured by half the intercepted arc, the Bragg angle of incidence (e_1) in Figure B1 is equal to half of angle a and the Bragg angle of diffusion (θ_2) equal to half angle b.

Since the angle of incidence is equal to the angle of diffraction,

$$26 = \frac{1}{2}a + \frac{1}{2}b$$
 (B1)

Since an arc is measured by the angle it subtends at the center of the circle,

$$a = \frac{180 \widehat{AB}}{\widehat{T}} \widehat{R}$$
and
$$b = \frac{180 \widehat{BC}}{\widehat{T}} \widehat{R}$$

Substituting for a and b in Equation (B1),

$$2 \theta = \frac{90 \ \widehat{AB}}{\Pi R} + \frac{90 \ \widehat{BC}}{\Pi R}$$
or
$$\theta = \frac{45}{\Pi R} (\widehat{AB} + \widehat{BC})$$
(B2)

From Figure 3, $\widehat{AB} + \widehat{BC}$ is found to equal $S_0 + S_1$, therefore,

$$\Theta = \frac{45}{\pi^R} (s_0 + s_1)$$
 (B3)

Using the following symbols as indicated,

 $\theta_{\rm p}$ = Bragg angle of incidence for platinum $\theta_{\rm m}$ = Bragg angle of incidence for martensite

R = radius of camera (Figure B1)

 S_p = distance from edge of film to platinum line (200) in mm. (Figure B1)

X = distance on film from martensite line to platinum line, in mm.,

and substituting in Equation (B3),

$$\theta_{\rm p} = \frac{145}{\pi R} \quad (s_{\rm 0} + s_{\rm p}) \tag{B4}$$

and

$$\Theta_{\rm m} = \frac{45}{\pi R} \quad (s_0 + s_p - x) \tag{B5}$$

Subtracting Equation (35) from (B4),

$$\Theta_{p} - \Theta_{m} = \frac{\mu_{5} X}{\mathbf{n}^{R}}$$
 (B6)

The radius (R) for the Phragmen No. 2 Camera is 50.5 mm. and the angle θ for the (200) plane of platinum using chromium Kal radiation is 35.717°. Substituting in Equation (B6),

$$35.717 - e_{m} = \frac{45x}{158.65}$$
or
$$e_{m} = 35.717 - .28364x,$$
(B7)

APPENDIX C

RELATION BETWEEN TETRAGONALITY AND INTERPLANER SPACINGS

Let

 d_1 = interplanar distance for the (110) plane of martensite,

d₂ = interplenar distance for the (101) plane of martensite. h,k, Miller Indices.

Starting with the equation for a simple tetragonal lattice,

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{\ell^2}{c^2}$$
 (C1)

Substituting (110) for (h,k,2),

$$\frac{1}{d_1^2} = \frac{2}{a^2}$$

$$a^2 = 2d_1^2$$
 (C2)

Substituting (101) for (h,k,2),

$$\frac{1}{d_2^2} = \frac{1}{a^2} + \frac{1}{c^2}$$

Substituting 2d₁² for a² (Equation C2),

$$\frac{1}{d_2^2} = \frac{1}{2d_1^2} + \frac{1}{c^2},$$

$$c^2 = \frac{2d_1^2 d_2^2}{2d_1^2 - d_2^2} \tag{C3}$$

Substituting a2 for 2d12,

$$c^2 = \frac{a^2 d_2^2}{2d_1^2 - d_2^2}$$
 (C4)

$$\frac{c}{a} = \sqrt{\frac{dz^2}{2d_1^2 - dz^2}}$$
 (C5)

APPENDIX D

EVALUATION OF TEMPERING PARAMETER N FOR EXPONENTIAL COOLING

To repeat Equations (3) and (4) of the text,

$$N = \int_0^t e^{-q/RT_{dt}} = \int_0^t t_{10} - 7340/T_{dt}$$
 (D1)

and

$$T = T_1 + (T_0 - T_1) e^{-ct}$$
 (D2)

Differentiating (D2),

$$dT = -c(T_0-T_1)e^{-ct}dt$$
 (D3)

$$dT = -c(T-T_1)dt (D4)$$

Substituting for dt in Equation (D1),

$$N = -\frac{1}{c} \int_{T_s}^{T_2} \frac{10^{-7340/T}}{T_{-T_1}} dT, \qquad (D4)$$

with the boundary condition N = 0 at $T = T_s$.

Then,

$$cN = \int_{T_2}^{T_S} \frac{10-7340/T}{T-T_1} dT.$$
 (D5)

Taking the temperature of the medium,

$$T_1 = 20^{\circ} \text{ C.} = 293^{\circ} \text{ K.}$$

and integrating down to,

$$T_2 = 30^{\circ} \text{ C.} = 303^{\circ} \text{ K.},$$

$$cN = \int_{303}^{T_s} \frac{10^{-7340/T}}{T_{-293}} dT \qquad (D6)$$

The ratio,

was then computed for various values of T as indicated in Table DI, and plotted as a function of T. By counting boxes, the area under the curve from 30° C. = 303° K. to T_{s} values of 100, 150, 200, 250, and 300° C., was determined*. This area represents cN. The results are given in Table DII and plotted in Figure 4.

^{*}Because the magnitude of the ratio varies so greatly, a separate plot with an appropriate scale had to be used for each of the $T_{\rm s}$ values.

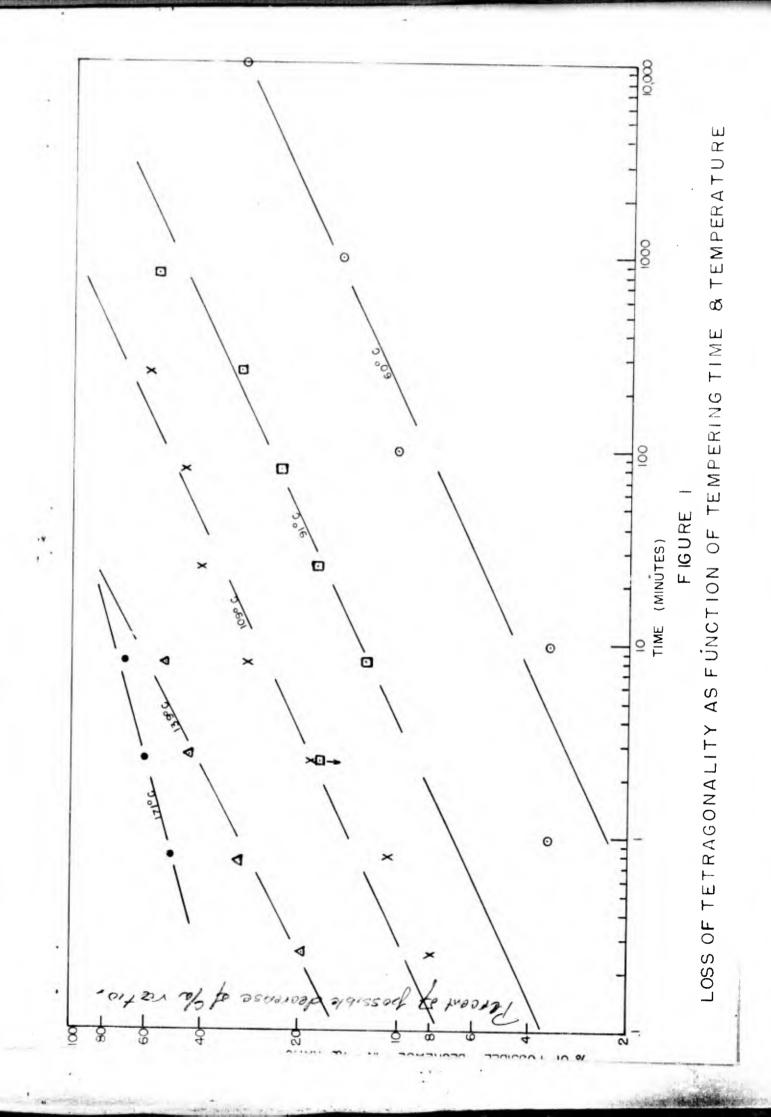
GRAPHICAL INTEGRATION FOR PARAMETER N ON EXPONENTIAL COOLING

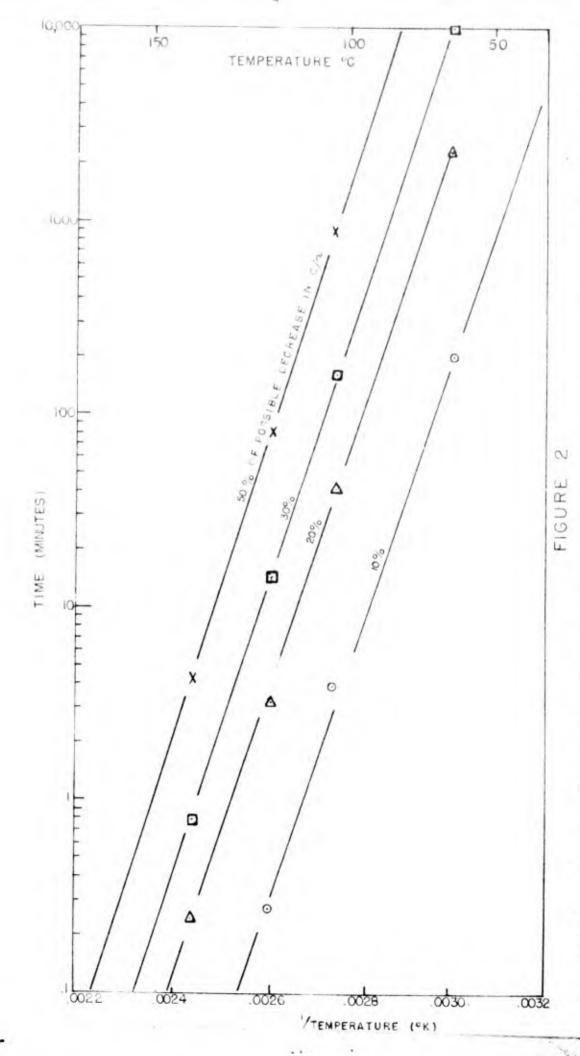
Temp.	°K.	7340/T	10-7340/T	T-293	10 ^{-7340/T} T-293
30 50 50 90 10 11 15 15 16 16 19 19 19 19 19 19 19 19 19 19 19 19 19	303 303 303 303 303 303 303 303 303 303	24.22 22.72 21.72 20.22 19.68 18.92 17.56 16.57 16.57 16.58 15.60 14.17 14.03 12.76 11.73 12.76 11.73 12.76 12.76 13.97 14.17 14.17 14.17 14.17 14.17 14.17 14.17 14.17 14.17 14.17 14.17 14.17 15.49 16.15	6.03x10-25 1.90x10-23 1.90x10-22 1.62x10-21 6.03x10-20 1.12x10-20 2.09x10-19 6.17x10-19 1.70x10-18 2.75x10-13 4.47x10-18 1.12x10-17 2.69x10-17 2.69x10-17 6.31x10-17 1.41x10-16 2.09x10-16 2.09x10-16 3.02x10-16 9.12x10-15 4.90x10-15 6.76x10-15 9.33x10-15 3.02x10-14 5.37x10-14 9.12x10-14 1.55x10-13 1.66x10-12 1.23x10-11 7.08x10-10 1.20x10-9	10 30 45 60 75 80 95 110 125 130 150 175 180 195 220 230 260 270 280 280 280 280 280 280 280 280 280 28	6.03x10-26 6.33x10-25 4.22x10-24 2.70x10-23 8.61x10-22 1.49x10-22 1.26x10-21 1.42x10-20 2.20x10-20 3.44x10-20 8.00x10-20 1.79x10-19 4.21x10-19 8.29x10-19 1.19x10-18 1.68x10-18 1.68x10-18 1.20x10-17 2.23x10-17 2.23x10-17 1.21x10-16 2.07x10-16 3.38x10-16 5.54x10-15 3.23x10-14 1.65x10-13 6.67x10-13 2.27x10-12

TABLE DII

RESULTS OF INTEGRATION

°C.	cN	log _{lO} cN
100	2.31x10 ⁻²¹	-20.64
150	3.75x10 ⁻¹⁹	-18.43
200	2.34x10-17	-16.63
250	6.48x10 ⁻¹⁶	-15.19
300	1.03x10 ⁻¹⁴	-13.99





TETRAGONALITY OF LOSS FIXED RELATION BETWEEN TIME AND TEMPERATURE FOR

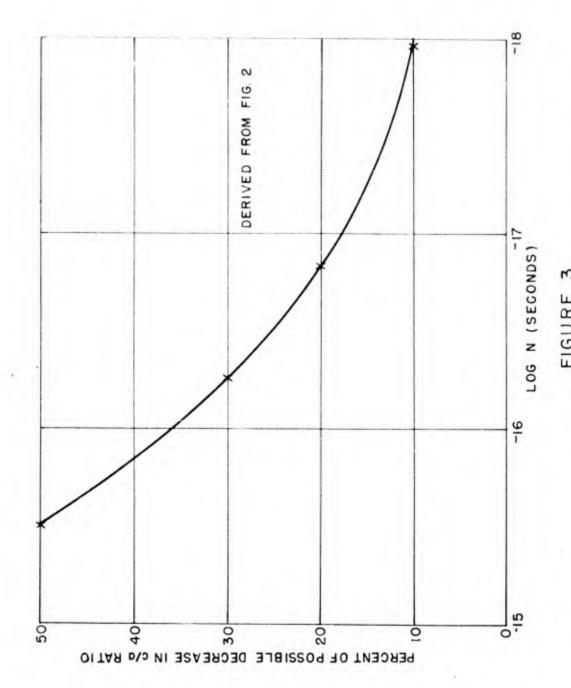


FIGURE 3 LOSS OF TETRAGONALITY AS FUNCTION OF TEMPERING PERAMETER N.

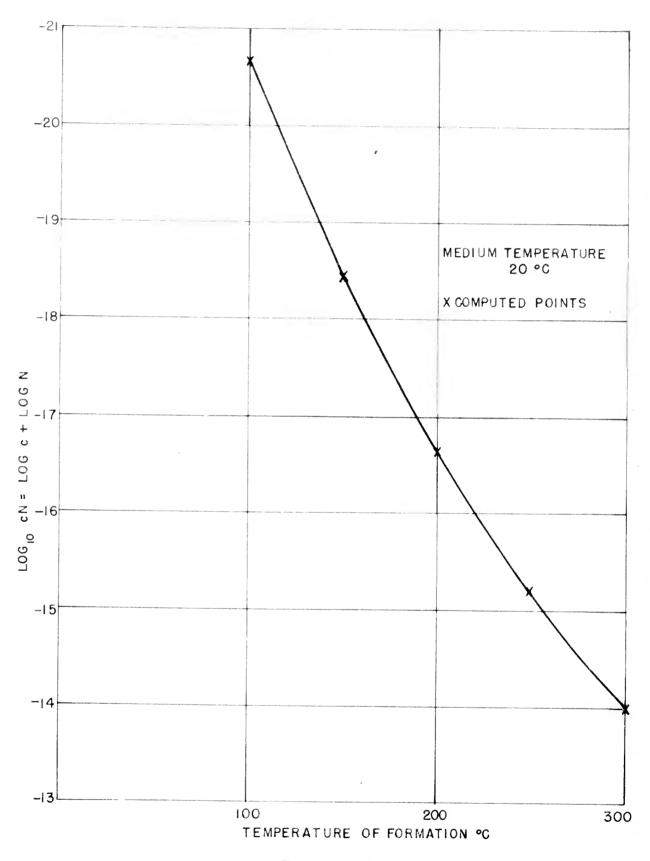


FIGURE 4

TEMPERING PARAMETER N FOR EXPONENTIAL COOLING AS FUNCTION OF TEMPERATURE OF MARTENSITE FORMATION & OF COOLING COEFFICIENT C.

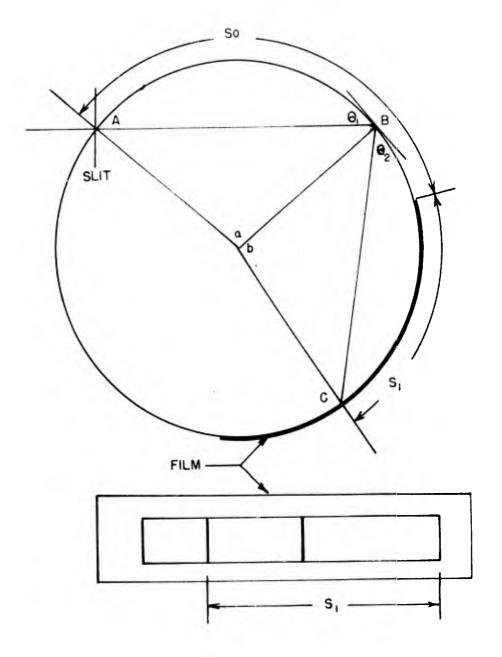


FIGURE BI CAMERA GEOMETRY